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AADE 2009-NTCE-11-04: LABORATORY METHODS TO ASSESS SHALE REACTIVITY WITH DRILLING FLUIDS

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Abstract

This paper describes laboratory methods used to assess shale reactivity with drilling fluids. These include descriptive techniques, various analytical techniques, and other test methods that employ standard laboratory equipment. Test methods and the respective equipment used include Sample Description, Thin Section Analysis (optical microscope), Scanning Electron Microscope (SEM), Cation Exchange Capacity (CEC), X-Ray Diffraction (XRD), Capillary Suction Test (CST), Swelling, Dispersion, Immersion, Bulk Hardness, Water Activity Measurement, and Fracture Development tests.

Types of shale samples include full-diameter cores, sidewall cores, wellbore cavings, and cuttings. The authors also present sample requirements for each method in terms of quantity, rigsite preparation, and shipping containers. A basic description, time required, and the data output for each method are explained.

Furthermore, how data from each method may be used to assess shale reactivity and help design shale inhibitive drilling fluid systems is briefly discussed. One observation is that methods that treat shale as a uniform bulk material without internal structure tend to be more “quantitative” while methods that treat shale as a non-isotropic material with internal structure tend to be more “qualitative”.

Introduction

The interaction between shale rock materials and drilling fluids is an important consideration when analyzing many drilling problems. Engineers use the term “shale” to describe a wide range of rock materials that are encountered in drilling operations. In order to perform meaningful tests that may help in selecting drilling fluid compositions that are less reactive with a particular shale interval, laboratory analysis of rock samples from that interval is useful.

In an ideal world, preserved full-diameter shale core would be available for analysis and testing. However, real world economics and practical drilling issues usually limit available samples to cuttings or cavings. To understand which analyses can be carried out with the available samples and, in some cases, to understand why obtaining better quality samples may be worthwhile requires understanding sample requirements for each type of analysis and test.

In planning and executing a meaningful program to test the interaction of drilling fluids with shale, the first step is to analyze the samples of shale that are available. There may be a tendency in engineering studies of shale to emphasize quantitative values such as CEC and semi-quantitative measures, such as percent mineral composition from X-ray diffraction. Perhaps, increasing awareness of qualitative aspects of shale from geological description, thin section, and Scanning Electron Microscope analysis can shed more light on the character of specific shale materials. Identification of features such as fractures, slickensides, consolidation state, bedding type, and thin layers of organic materials contribute to understanding how fluids react to destabilize shale.

Both qualitative and quantitative measures of shale characteristics can be used to informally classify shale as having high, moderate or low reactivity. Drilling problems can occur in all classes of shale. The typical nature of drilling problems encountered and the type of shale-fluid testing that is most meaningful generally is determined by the reactivity class of the shale. The objective of this approach is to determine the type of test program likely to be related to the drilling problems encountered; to provide help in selection of inhibitive fluids is the goal of this approach.

Sample Description

Description of the shale is possible with any size sample including cuttings, cavings, sidewall cores, and full-diameter cores. The equipment used for description includes hand lens and simple stereomicroscope. For non-geologists, a modest amount of training and supervised practice may be required. Basically, the technique involves careful examination of the sample for at least a few minutes and identifying the following:

- Bedding structure
 - Current deposited: cross bedding, wavy bedding, flaser bedding, starved ripples, ripples¹
 - Laminations
 - Graded bedding
 - Sandstone or siltstone interbeds
 - Disturbed bedding
 - Massive: lack of bedding structure
- Sedimentary structures
 - Burrows
 - Sole marks
 - Load casts
 - Mud cracks
- Color – Common shale colors: white, brown, gray, tan, red, purple, green, and black.
- Fractures
 - Parallel to bedding or laminations
 - Shear fractures
 - Cracks
 - Slickensides
- Consolidation and state
 - Plastic and not sticky
 - Plastic and sticky
 - Friable
 - Firm
 - Hard

Usually, descriptive information is qualitative rather than quantitative. Nevertheless, the presence of features, such as natural fractures or slickensides, may offer important clues about the causes of wellbore instability. Sand or silt interbeds may provide a pathway for fluids with

the potential to swell clays, to reach into the rock behind otherwise impermeable shale.

X-ray Diffraction

X-ray Diffraction (XRD) is an instrument used to identify the minerals present in shale samples. The sample is placed into the instrument and rotated through a series of angles while being illuminated with the X-ray beam. As the sample is rotated in the instrument, the crystalline structures of the individual minerals present diffract the X-ray beam. This results in an X-ray diffraction pattern that is unique for each mineral in the sample. The software identifies the minerals present and determines semi-quantitative amounts of each.

X-ray diffraction can be carried out on cuttings, cavings, or cores. Preferably, samples should be washed to remove drilling fluids at the well site before being sent to the lab. Once at the lab, samples from water-based fluids may require some further washing to remove any residual drilling fluid. If the cuttings are not solid and rather soft and mushy, the sample is dried as received without washing.

Conversely, samples from non-aqueous fluids are cleaned with organic solvents to remove hydrocarbons and dried. Once the samples are dry they are ground to a fine powder and placed in a sample holder for analyses. The time required for the instrument to complete the X-ray diffraction pattern is two minutes. The dry powder analyses, or bulk samples analyses, can be supplemented by a clay fraction analysis, which is achieved by separating the clay fraction from the bulk sample. A dispersion of the powder in water is prepared, the coarse non-clay fraction is allowed to settle, and the resulting clay suspension is filtered. While still wet, the filter cake of the clay fraction is transferred to a glass slide and air dried. X-ray diffraction of the clay slide results in an enhanced analyses of the clays present. The presence of smectite clays can be further enhanced by treating the clay slide with glycol.

X-ray diffraction analyses can be done using a very small amount of about one gram of dried solids, but 100 g should be submitted if the sample requires washing and or other testing. An X-ray diffraction analysis requires expensive instrumentation and a knowledgeable analyst trained in the operation of the instrument and interpretation of the data.

Owing to limitations of obtaining pure standards and the crystalline nature of some samples, the X-ray diffraction data is only semi-quantitative for the mineralogical composition of the shale even though advances in the use of Rietveld software routines for quantitative analyses have made improvements. The non-reactive minerals seen typically in shale samples are quartz, feldspars, calcite, dolomite, pyrite, hematite, and siderite, while the reactive clay minerals seen are illite, kaolinite, chlorite, smectite and mixed layer clays. The smectite and mixed-layer clays are the most reactive and are prone to swelling.

The higher the clay content, the more likely the shale will be reactive to swelling. Therefore, the X-ray diffraction data can be used in conjunction with other considerations when formulating a drilling fluid for specific sections of the well. When it is known that a section of the well will have high clays present, a more inhibitive drill fluid should be considered.

Cation Exchange Capacity

The Cation Exchange Capacity (CEC) is a measure of the exchangeable cations present on the clays in a shale sample. These exchangeable cations are the positively charged ions that neutralize the negatively charged clay particles. Typical exchange ions are sodium, calcium, magnesium, iron, and potassium. Most of the exchangeable ions in shale samples are from the smectite (bentonite, montmorillonite) clays. The CEC measurements are expressed as milliequivalents per 100 g of clay (meq/100 grams).

Typically, the oil and gas industry measures the CEC with an API-recommended methylene blue capacity test.² Other methods for determining CEC also are available, such as a colorimetric technique based upon cobalt hexamine trichloride depletion³; an ammonium acetate saturation method⁴; and copper complexes methods⁵ among others. The API-recommended methylene blue test (MBT) requires one gram of finely ground dried shale. The sample is dispersed in water with a small amount of dispersant, sulfuric acid and hydrogen peroxide; boiled gently for a few minutes; cooled to room temperature; and titrated with a methylene blue solution. The end point is reached when a drop of the sample suspension placed on a filter paper results in a faint blue halo surrounding the dyed solids. The CEC analyses can be determined in a laboratory or at the well site with a minimum amount of equipment.

The higher the CEC is, the more reactive the shale. Sandstone and limestone typically are nonreactive and have CEC values of less than 1 meq/100 g. Moderately reactive shale has a CEC value from 10 to 20 meq/100 g, while reactive shale has a CEC value greater than 20 meq/100 g. A low CEC can still be problematic if the small amount of clays present swell and cause the shale to break apart. A higher CEC shale sometimes is referred to as “gumbo shale.”

Typical CEC values for various clays found in shale and sand are:

Smectite	80 to 120 meq/100 g
Illite	10 to 40 meq/100 g
Kaolinite	3 to 15 meq/100 g
Chlorite	10 to 40 meq/100 g
Sand	<0.5 meq/100 g

Thin Section Analysis

Thin section analysis, a standard geological method, requires a transmitted light microscope. Specialized “Petrographic” microscopes are transmitted-light microscopes equipped with a rotating stage and a polarized light analyzer to permit measurement of optical properties of crystalline materials. Thin sections are standard 30-micron thick sections of rock mounted on a glass slide.

Thin section analysis requires samples that can be sawed or sectioned. Full-diameter core and many, if not most, sidewall core samples can be sectioned. Further, cavings generally can be sectioned along with large and firm cuttings. Samples that have been reasonably preserved and not dried out are preferred for sections since drying clays can induce drying cracks in many shale samples. Samples 1-cm in size or larger are needed for thin sections. The thin section is examined systematically using the transmitted light microscope. Key features that can be identified from thin section are:

- Texture class
 - Claystone
 - Mudstone
 - Siltstone
- Mechanical support structure of the shale
 - Grain supported
 - Clay supported
 - Mineral cement supported
- Microfractures and pores
 - Presence and number of microfractures
 - Orientation of microfractures – parallel or intersected
 - Width of microfractures
 - Macropores – present or absent
- Organic Materials
 - Presence of mechanically weak organic-rich layers
 - Organic materials dispersed in the shale

The key information obtained from thin sections often is qualitative. Understanding the mechanical support structure of the shale may identify zones more likely to fail because of inadequate mud weight. Microfractures can provide conduits for fluids to enter the shale and provide failure planes for stress-induced slippage and sloughing. Organic materials that are oil-soluble or that soften when absorbing oil can make otherwise non-reactive shale sensitive to oil-based fluids.

Scanning Electron Microscope

Scanning Electron Microscope (SEM) is a tool for performing high-magnification analyses of shale. The SEM allows for better three-dimensional observations of micro-fractures and cavities in the shale that are not easily seen using transmitted light or transmitted electron microscope techniques. The texture and orientation of the shale, its degree of compaction, and the presence of imbedded minerals and pores can be observed. Any invasion of drilling fluid through the microscopic pores also can be demonstrated using the SEM. The addition of an energy dispersive X-ray fluorescence detector (EDAX) to the SEM enhances the analyses by showing the chemical composition of selected areas. Chemical analyses is the relative amounts of each element, including Si, Al, Na, K, Ca, Mg, Fe, O, Ba, and S that are present in the selected area being analyzed. This chemical analysis makes it possible to further identify the minerals under observation by the SEM, such as clays, quartz, feldspars, calcite, and iron minerals, or if any barite is present that could indicate drilling fluid invasion into the pores or fractures. The ability of the EDAX to show values for carbon and oxygen aid in the identification of organic material that might be imbedded in the sample.

SEM analysis can be carried out on cavings, cores, or relatively large pieces of cuttings. Ideally, the sample needs to be large enough to be cut or broken to expose a fresh surface for analyses in order to see any clean pores or fracture planes that have not been exposed to drilling fluid. Minimum sample preparation is required with the clean sample being mounted on a small stage and placed into the instrument. Magnification ranges used for shale analyses typically range from 20 to 500X, whereas most instruments can achieve much higher magnifications with some as great as 1,000,000X. The SEM analyses can be quite time consuming, depending on the operator and what is being observed. This analysis requires expensive instrumentation that is not available in most laboratories and a knowledgeable analyst, who is trained in the operation of the instrument and interpretation of both the digital images and the chemical analyses associated with the images.

SEM photographs and chemical analyses data are qualitative tools used in the evaluation of the overall nature and consistency of the shale. This evaluation comprises its composition; the presence, size, and shape of pores; what minerals are holding the sample together; the minerals that might be imbedded in the sample matrix; and what minerals in the sample and where they are located that might be subjected to swelling and result in dispersion when exposed to fluids. Limitations to SEM analyses are selecting the right samples to examine that would be representative of the formation of concern and recognizing texture and fabric characteristics too large to identify with SEM.

Water Activity

Measurement of water activity in shale is the basis for running balanced-activity oil-based mud. The adsorption isotherm method to measure water activity in the lab uses laboratory desiccators with saturated salt solutions that regulate the vapor pressure of water. A second method uses a hygrometer to measure the relative humidity of an enclosed space containing the cuttings. The percent relative humidity divided by 100 closely approximates the water activity.

Wellsite sample collection, treatment, and preservation of the shale is very important in obtaining suitable samples for water activity measurement. The samples can be cuttings, cavings, or cores drilled with non-aqueous drilling fluid. Cuttings or cavings need to be washed with base oil to remove any oil or synthetic-based drilling fluid that adheres to the samples. After the samples have been washed with base oil, it is essential for the samples to be sealed in air-tight containers, since shale exposed to the atmosphere slowly equilibrates with the activity of water or relative humidity in the atmosphere. Approximately 100 g of cuttings or cavings are needed for analysis.

In the water-adsorption isotherm method, percent water is plotted against water activity. The percentage of water in the sample correlates to a specific activity on the adsorption or desorption isotherm.⁶ Special hygrometer kits that include sample flasks, adapters, and calibration aids are available for measuring shale water activity. In addition, the hygrometer can be used to measure water activity of the oil-based drilling fluids. Although room temperature measurements of water activity are straightforward, measurement of water activity at high temperature, high pressure, and mechanical stress present significant challenges.

Swelling Test

The swelling test is conducted using the linear swelling tester. This device measures free swelling of a reconstituted shale pellet after the shale has been in contact with a drilling or completion fluid. The amount of swelling the shale undergoes after it is in contact with the fluid is a measure of the reactivity of the shale to the fluid.

The device consists of a fluid reservoir, a shale chamber, a linear variable differential transformer (LVDT), A/D converter, and computer. The fluid reservoir holds the fluid until the test is ready to start. The shale pellet, which is prepared by compressing dry ground shale under 25,000 psi, is placed in the shale chamber that confines the pellet between a pair of screens and confines swelling to the vertical direction. The fluid is released from the reservoir and fills the shale chamber, thus coming into contact with the shale as the test begins. The swelling of the shale causes the LVDT sensor to rise, altering the inductance of the transformer and generating a voltage change which is

sensed by the A/D converter. The A/D converter status is monitored by a computer at short intervals and the results, in terms of percent volume expansion of the shale, are recorded for the duration of the test.

This test is a good indication of the reactivity of the shale sample to the fluids being tested. For comparison, a shale pellet is exposed to water in order to determine the worst-case scenario. The more swelling observed with water, the higher the content of swelling and highly water-sensitive clays (smectite and mixed-layer clays). This test can be performed on cuttings, cavings, sidewall core, and full-diameter core, providing the amount of sample is sufficient to prepare one pellet for each fluid to be tested. It is important to note that the preparation of the sample implies disintegration and reconstitution of the sample into a pellet. Formation samples with massive structures and homogeneous composition are most suitable for this test. The results of the test are associated mainly with a chemical interaction and some physical effects, such as fracturing, dispersion and cracking, are not detected due to the elimination of the natural structure of the rock.

Dispersion Test

This test is used to design fluids and screen the effectiveness of inhibitor additives to maintain the integrity of the cuttings and minimize the interaction of fluids with the shale sections during the drilling and completion operations. Medium to high reactivity shale formations with high to fairly high amounts of smectite and moderate content of illite tend to be suitable for this test. Cuttings recovered from wells drilled with oil-based fluid are the most common samples used for this test. Cuttings recovered from water-based fluid are not recommended, because the shale may have already dispersed and reacted before being used for the actual test. Cavings, sidewall core and full-diameter core may also be used for this test.

The dispersion test involves exposing a weighed quantity of sized shale pieces (-6 mesh and +20 mesh) to a formulated fluid in a conventional roller-oven cell.⁷ The test provides long-term exposure of the shale to the fluid under mild agitation. Under such conditions, dispersion of the shale into the fluid will occur depending on the tendency of the shale to disperse and the inhibitive properties of the fluid. The rheological characteristics of the fluid also can influence the test results by altering the amount of agitation in the rolling phase. The fluid and shale are rolled together in a roller oven for 16 hours at 150°F. After cooling to room temperature, the fluid is poured over a 50-mesh sieve and the retained shale pieces are recovered, washed, weighed, and dried overnight at 210°F. Afterwards, the sample is re-weighed to determine the percent recovery.

Capillary Suction Time Test

The Institute of Water Pollution Control in the UK originally used the capillary suction time (CST) device to measure the time required for a slurry filtrate to travel a given distance on thick porous filter paper.⁸ This technique has been adapted to measure the capillary suction time of clay or shale slurries. Basically, a small amount of shale is mixed with water, brine, or mud filtrate in a small commercial blender cup.

The CST requires 3 g of dry cuttings, cavings, or core material per test. Typically, several tests are run at different salt concentrations, and it is common to run repeat tests with the output comprising of numerical results in seconds. In many cases, a “flocculation” concentration of salt or KCl that dramatically reduces CST can be determined. If the salt

concentration in the fluid is lower than the flocculation concentration, particles of shale will disperse into the fluid.

Reactive shale with high smectite clay content usually has a high CST value less reactive shale lacking smectite clay tends to have a significantly lower CST value. In addition, capillary suction time can be used to evaluate the effect of salinity on shale dispersion tendencies for specific shale formations.

Bulk Hardness Test

The bulk hardness test is designed to evaluate the hardness of shale after exposure to fluids. The hardness of the shale can be related to the inhibitive properties of the fluid being evaluated. Shale that interacts with the fluids will become softer due to the adsorption of water, swelling, and dispersion of fine particles. This rock-fluid interaction can be linked to wellbore stability problems, including reduction in the compressive strength, spalling or fracturing. In terms of integrity of drill cuttings, excessive softening and stickiness of the pieces of shale can produce mud rings in the annulus, sticking problems in the drilling assembly, and bit balling among other problems.

In this test, sized shale pieces are hot rolled in the test fluid for 16 hours at 150°F. After hot rolling, the shale pieces are recovered on a 50-mesh sieve and placed into the bulk hardness tester. Using a torque wrench, the shale is extruded through a perforated plate, measuring the maximum torque required for each turn in compression. Depending upon the condition of the cuttings, the torque may reach a plateau region or continue to rise during the extrusion. Harder and more competent shale pieces will deliver higher torque readings. Cuttings are the most common samples used for this test. Cavings, sidewall cores and full-diameter cores also are suitable samples for conducting the bulk hardness test.

Fracture Development Test

The Fracture Development Test identifies and tracks the development of fractures in shale formations when they are exposed to drilling fluids.⁹ This test focuses on low reactivity/hard shale formations where stability problems are related to propagation of pre-existing fractures and development of new fractures. Observations made during exposure of shale to fluid are photographically documented. The samples are carefully examined with a microscope after fluid exposure to document the development of fractures and other changes that occur in the samples. Quantitative statistics and photographs documenting fracture development are obtained. The test documents stability and failure of shale in different fluid environments. The fluids used for this test can be clear/solids-free (base of the drilling fluid plus inhibitor products or completion fluids) or fluid systems containing solids.

Two techniques are used for this purpose: Time-Lapse Photographs (TLP) and microscope analysis of thin sections.

Shale exposure to fluid: If clear fluids are used, the TLP technique is used to record photographically the visible changes in the rock during fluid exposure over a period of time (usually 24 hours).

Microscope analysis technique is used to observe the changes in the micro-structure of the rock using thin sections of the shale samples after fluid exposure. Photomicrographs of the sections are taken to show fractures present. Measurements are recorded to compare

quantitatively the effects of the fluids (*e.g.*, maximum fracture width, number of fractures, typical fracture width).

The petrographic studies of thin sections of shale sample exposed to fluids facilitate the identification of areas where the rock structure fails or becomes weak. Fractures are more likely to occur along pre-existing micro-fractures, laminations, slickensides, and boundary areas.

Shale core samples are needed for the fracture development test. Usually a piece of rock is taken from the core and it is sawed into small, similar sized pieces (cubic or cylindrical shape). For testing, the maximum sample dimension (length or diameter) may vary depending of the size of the core and the rock conditions but in general terms the maximum dimension ranges between 1 to 2 inches. Core samples must be in good conditions and preserved samples, typically cores that are sealed in wax, are the most suitable for this test. Preserved samples tend to maintain their natural wettability and the fracture network is conserved and less likely to be altered by the natural drying process. Samples that have been allowed to dry or exposed to the air are unsuitable for this test. Full-diameter core or core plugs that may be available from commercial core testing companies can be used for this test. Typically four to six fluids can be tested with a 6-in. full-diameter core sample.

Immersion Test

The Time Lapse Photography (TLP) technique can be used with medium reactive shale samples to identify, track, and describe in a qualitative manner the instability mechanisms of rock samples when immersed in various fluids. Photographic documentation of the behavior of the rock can show simultaneous mechanisms of rock-fluid interaction, such as dispersion of fines, spalling of fragments, cracking along weak planes, boundary areas or laminations. After exposure to the fluids, the samples usually are fragile or may fall apart easily.

Discussion

Different laboratory methods are used to assess the reactivity of the shale formations with fluids. Several methods are used to estimate the reactivity of shale formations based on the analysis of the rock samples. Other methods are used to evaluate the reactivity of the shale based on test procedures that involve the direct interaction of the rock samples with fluids.

In many cases, the analysis of the shale sample is limited to XRD analysis, which is used widely to identify the presence of clay minerals. In addition, the Cation Exchange Capacity (CEC) is used frequently as an indicator of the reactivity potential of the shale sample. These two methods are used extensively to categorize the reactivity of shale formations and anticipate the need for an inhibited drilling fluid. However, a more complete description and analysis of the sample can help to identify other features of the shale samples that are relevant in the stability of these formations. Visual examination, thin section observations, and SEM analyses can be used to identify particular characteristics of the samples in terms of texture, structure, consolidation, fracturing, etc. These characteristics can be used not only to recognize the type of shale formation, but also to understand potential instability mechanisms.

If the characteristics of the shale samples are linked with instability mechanisms such as swelling, dispersion, fracturing, softening, etc., the

test selection becomes a more logical process that allows examiners to obtain meaningful data and also reduces laboratory time. The shale stability tests with fluids are designed to evaluate particular mechanisms of instability. The results of these tests reflect the effectiveness of the fluids to control and minimize a particular instability behavior.

Figure 1 presents a chart summarizing the shale sample test selection based on common characteristics of the shale obtained from preliminary analysis. Description and analyses of the rock samples constitute the first step of this process. The results of these analyses provide information to classify the sample according with the characteristics. Three groups of shale samples are presented: High reactivity, moderate reactivity, and low reactivity shale. The characteristics listed for each group include aspects such as composition, structure, description, consolidation state, and CEC values. These groups of characteristics cover the common features of shale samples that can be obtained using relative standard techniques. The testing methods to evaluate the reactivity of shale with the fluids are listed below each group according to mechanisms of the instability expected for the samples. The sample preparation process was also considered in selection of tests for each group.

The impact of the sample preparation process of each test needs to be well understood. Some of the preparation processes vary from preservation of the rock integrity to total destruction of rock structure. Formation samples with massive structures and homogeneous composition may be tested differently than the samples with moderated and highly laminated structures. The samples with massive structures and lack of significant variations in composition can be ground to a fine powder or size into small fragments (<3 mm) without a significant impact on the interpretation of the test results. These samples generally are highly swelling and dispersive type of formations that can be used for swelling, capillary suction time and dispersion test. The highly laminated and hard shale formations exhibit clear variation in composition and structure along the bedding planes. It is important to maintain the integrity of the rock and use a testing technique to evaluate the propagation and development of fractures which is the common mechanism of instability observed in this type of shale.

Fracture development is a technique specially designed to maintain the shale integrity and evaluate the fracturing behavior of low reactive formations. The moderately reactive shale exhibits characteristics that can be evaluated using techniques that partially maintain the integrity of the sample and evaluate common instability mechanisms of this type of formation. Dispersion test, bulk hardness and immersion tests are the techniques typically used for this kind of shale.

Some of the most frequent limitations to a specific testing plan are the quality and amount of sample. Table 1 shows the recommended amounts of sample required to conduct the shale analysis and the test with fluids.

Conclusions

The integration of different aspects in the shale analysis constitutes an important approach to assess the reactivity of shale with drilling fluids. The use of different methods to observe and describe, in a qualitative manner, significant features of shale formations can expand the comprehension of the interaction of shale formations and fluids.

The quantitative and semi-quantitative methods, frequently used in the analysis of shale, can be used in combination with qualitative methods to obtain a more complete characterization that contains key elements to interpret and understand the behavior of shale formation in drilling fluids. From the shale analysis information, a general classification of the shale can be used to categorize the reactivity and anticipate the potential instability mechanisms with fluids.

The selection of tests to evaluate the rock/fluid interactions becomes a more logical process if valuable information about the samples is obtained prior to the testing. Extensive and time-consuming testing programs can be reduced significantly if fundamental information is obtained directly from the shale samples. The adequate use of this information can steer the laboratory efforts in the process of finding solutions to the instability problems in the drilling operations.

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Table 1 – Recommended Amounts of Sample Required to Conduct the Shale Analysis and Tests with Fluids

Test	Type of sample	Amount per Test
X-Ray Diffraction	Cutting, cavings, cores	5 grams of clean dry sample
Cation Exchange Capacity	Cuttings, cavings, cores	1 gram of clean, cry, ground powder
Scanning Electron Microscope	Cuttings, cavings, cores	1 gram
Water Activity	Cuttings, caving, core	100 grams
Thin Sections	Large cuttings, cavings and core material	Samples approximately 1 cm or larger
Swelling Test	Cuttings, cavings, core material	5 grams ground powder
Capillary Suction Time Test	Cuttings, cavings, core material	3 grams ground powder
Dispersion Test	Cuttings, cavings, core material	20 grams (-6 mesh and +20 mesh)
Bulk Hardness Test	Cuttings, cavings, core material	30 grams (-6mesh and +20 mesh)
Immersion Test	Cavings, core material	Similar sized pieces (cubic or cylindrical shape). Sample dimension (length or diameter) ~ 1-2 inches
Fracture Development Test	Core material	Similar sized pieces (cubic or cylindrical shape). Sample dimension (length or diameter) ~ 1-2 inches

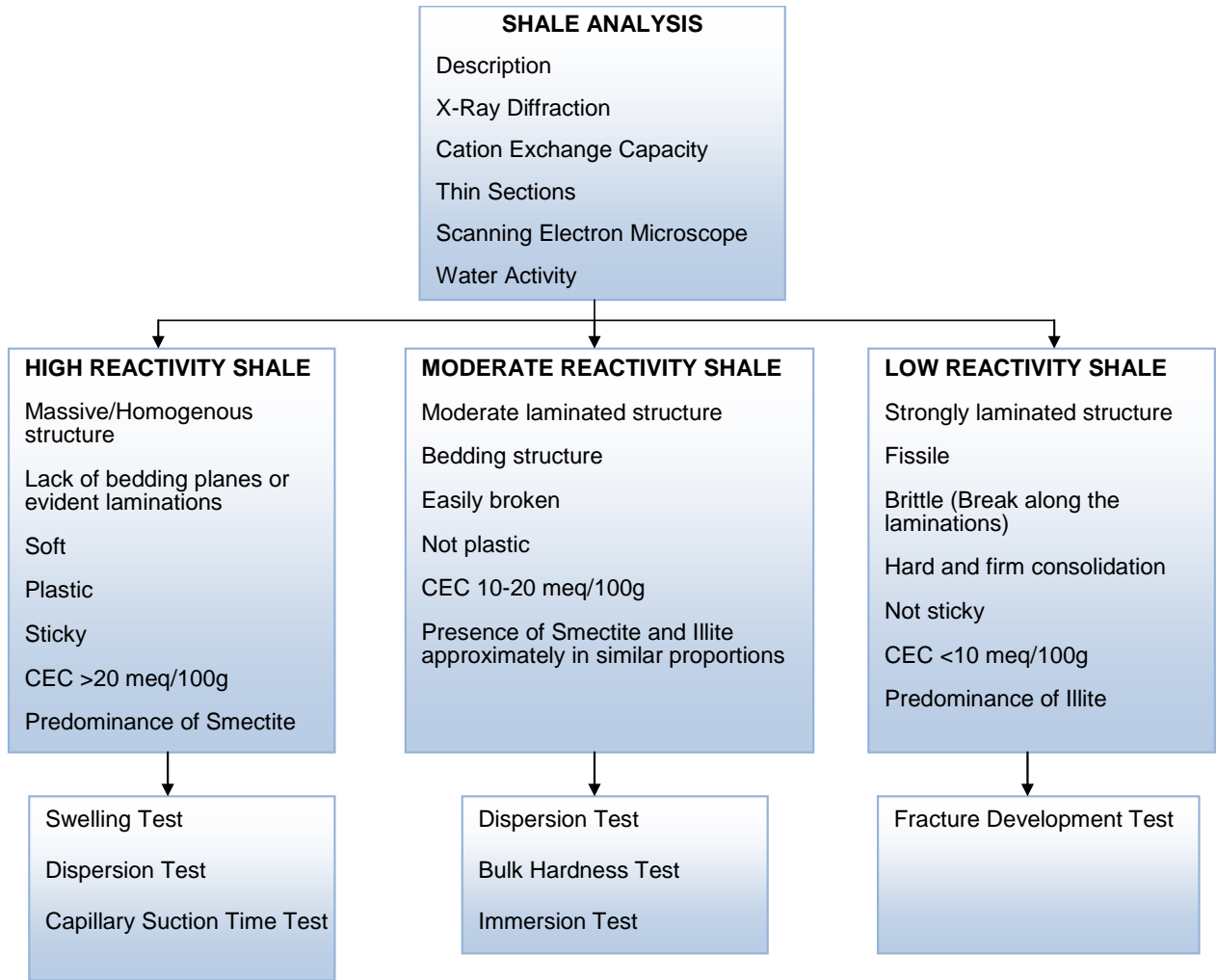


Figure 1 – Test selection to assess the reactivity of shale with drilling fluids based on common characteristics of shale samples.